IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants:	Alexander P. Moravsky, et al.	**Examiner:**	Ashok Patel
Serial No:	09/680,291	**Art Unit:**	2879
Filed:	October 6, 2000	**Docket:**	21088/14311
For:	DOUBLE-WALLED CARBON NANOTUBES AND METHODS FOR PRODUCTION AND APPLICATION		

Confirmation No.: 9193

Mail Stop Amendment
Commissioner for Patents
P.O. Box 1450
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DECLARATION OF ALEXANDER P. MORAVSKY

I, Alexander P. Moravsky, hereby declare and state as follows:

1. I am one of the applicants of the above-identified application, and I have complete knowledge of all aspects of the application.

2. I am currently employed as Senior Scientist at MER Corporation, Tucson, Arizona, and have been so employed since 1999. Previous to that year, I assisted MER Corporation in Tucson, Arizona for several months in 1998 as a consultant. My employment history since 1975 is set forth in the Resume of Alexander P. Moravsky, attached hereto as Exhibit 1 and incorporated herein by reference and made a part hereof.

3. I am currently involved in fullerene and carbon nanotube materials synthesis and property studies, and have been so involved since 1991. I have extensive experience and many publications in this field, as set forth in my attached Resume in Exhibit 1.

4. I have earned Ph.D, M.Sc. and B.Sc. degrees in the fields of Chemistry, Physics and Molecular and Chemical Physics, all as set forth in my attached Resume in Exhibit 1.

5. The present application is directed to, *inter alia*, a solid substance comprised by more than one half by weight of hollow carbon nanotubes having walls consisting essentially of two layers of carbon atoms, said nanotubes consisting of two concentric nearly cylindrical graphene layers.

6. I have been advised by counsel that the United States Patent and Trademark Office ("USPTO") has considered the following claims as patentably distinct from the subject matter described in the previous paragraph:

> 108. Substantially pure double-wall nanotubes, whereas the double wall nanotubes have two concentric nearly cylindrical graphene layers.
>
> 109. The double-wall nanotubes according to Claim 108 which additionally contain single-wall nanotubes, wherein the ratio of double wall nanotubes to single wall nanotubes is greater than 30.
>
> 110. The double wall nanotube according to Claim 108 wherein the outer diameter of the double-wall nanotubes ranges from 3.4 to 5.5 nm, and the inner diameter ranges from 2.7 to 4.7 nm.

7. The subject matter described in Paragraph 5 encompasses the subject matter in Paragraph 6. More specifically, the subject matter in Paragraph 5 is directed to a solid which is comprised of more than 50% by weight of double wall nanotubes, where the nanotubes consist of two concentric nearly cylindrical graphene layers, while Claims 108-110 comprise substantially pure double wall nanotubes, where the double wall nanotubes have two concentric nearly

cylindrical layers. Thus, a product, which is substantially pure double wall nanotubes, as recited in paragraph 6, is encompassed by the subject matter in Paragraph 5.

8. I have also been advised by counsel that the USPTO in the latest Office Action has cited U.S. Patent No. 6,743,408 to Lieber et al. ("Lieber et al."). I have been advised by counsel that Lieber et al. filed a provisional application on **September 29, 2000** to which it is claiming priority. For purposes of this Declaration, it is assumed that September 29, 2000 is the effective filing date of Lieber et al.

9. The present application was filed in the USPTO on October 6, 2000, which date is approximately 7 days after the earliest filing of Lieber et al.

10. The invention described and claimed in the above-identified application was completed in the U.S. prior to September 29, 2000.

11. As evidence hereof, annexed hereto and made a part hereof are Exhibits 1-11. All of the acts described herein took place in either the Untied States or a NAFTA country other than the United States or a WTO member country prior to September 29, 2000, and were performed by myself or by scientists and/or technicians working under my direct supervision or control, except to the extent indicated herein. Data or information not pertinent to the invention and dates have been masked out in the preparation of their photocopies.

12. Attached hereto as Exhibit 2 is a copy of a notebook page, which outlines a synthesis for a product of the present invention. This outlines an arc discharge evaporation of a metal-graphite electrode, where the anode was a graphite rod (8.2 mm in diameter), having a drilled channel of about 189 mm in length and 3.16 mm in diameter. The cathode was a graphite rod, which was positioned coaxially with the anode. The catalyst was prepared by heating nickel, cobalt and iron together with elemental sulfur. The resulting conglomerate was ground in

a ball mill to a micron particle size and then intimately mixed with carbon powder. The channel drilled in the graphite rod was tightly filled with the mixture to obtain an anode containing 79.91% C, 10.26% Ni, 2.77% Co, 5.44% Fe and 1.62%S.

An arc discharge was carried out with an arc current of 81A at 350 Torr in an atmosphere of Argon (175 Torr) and hydrogen (175 Torr) (1:1). The voltage drop across the gap was 26-28 V, and the rod feed was 2 mm/min. The duration of the arc process was about 45 min.

The carbon material produced in the arc discharge was deposited on the walls of the reactor and on the electrodes wherefrom it was collected separately. A fibrous material (elastic web-like product) was collected from the cathode.

13. A SEM examination of the web-like products obtained from the process described in Paragraph 12 showed dominating amounts of curled fibers being present in the product.

14. Exhibit 3 is an example of a TEM image of the product obtained from the process described in Paragraph 12. As shown in Exhibit 3, unusually thick nanotubes of approximately 4 nm in diameter were observed, assembled in thin bundles containing a few tubes.

15. The image was quite different than the appearance of single walled nanotubes, commonly produced in arc discharge processes, whereby tubes are about 1.5 nm in diameter and the bundles contain some dozen tubes.

16. Further, the walls of the tubes in Fig. 3 are thicker than is observed for commonly produced SWNTs, which is consistent with the nanotubes having two walls.

17. TEM images made by an instrument with better resolution unambiguously show that the tubes produced by the process described hereinabove in paragraph 12 have a two-wall structure. The images are attached hereto as Exhibits 4-8. These images unambiguously show that the nanotubes have two concentric nearly cylindrical graphene layers.

18. Additional samples were prepared in accordance with the procedure outlined in Example 1.

19. Samples, which were prepared by me or under my direct supervision, in accordance with the procedure described in the underlying application, such as Example 1, were examined under HRTEM (High Resolution TEM) in Russia by my colleague, Dr. Krinichnaja. Attached as Exhibit 9 is an email message from Dr. Krinichnaja in Russian; Exhibit 10 is a translation thereof, which verifies that the sample which I provided to her and which she examined using TEM, consisted of double wall nanotubes, having diameters of 3.5 to 5.2 nm. Good quality HRTEM analysis of the samples produced prior to September 29, 2000 revealed that samples contained not only DWNTs as the dominant carbon product, and also that no SWNTs were detected.

20. As further evidence, I enclose a copy of an article authored by, *inter alia*, Dr. Loutfy and myself that was published in Carbon 39, 2001, 761-770, which is attached as Exhibit 11. Although the article was published in 2001, it was received by the publisher on June 2, 2000, and accepted for publication on July 24, 2000, both dates of which are prior to the September 29, 2000 date of the earliest filing of Lieber et al. All of the experiments and the data described in the paper referred to in Exhibit 11 were conducted prior to the September 29, 2000 date of the earliest filing of Lieber et al.

21. Although the paper in Exhibit 11 mainly refers to a HRTEM study of DWNTs, it also provides a description of the preparation of DWNTs. The preparation of the nanotubes was conducted by Dr. Loutfy and/or myself or under our direct supervision, and the synthesis is in accordance with the procedure we described in our underlying application. As shown by the

HRTEM micrographs, the synthesis produced double wall nanotubes having two concentric nearly cylindrical graphene layers.

22. As shown by all the data described herein, all of the experiments described herein produced a solid product comprised by more than one half by weight of hollow carbon nanotubes having walls consisting essentially of two layers of carbon atoms, consisting of two concentric nearly cylindrical graphene layers. All of the experiments described herein were performed prior to September 29, 2000.

23. Thus, the sample of solid product described in the underlying application which is comprised of more than one half by weight of hollow carbon nanotubes having walls consisting of two layers of carbon atom, and consisting of two concentric nearly cylindrical graphene layers were synthesized and analyzed prior to the September 29, 2000, the earliest filing date of Lieber et al.

24. I further declare that all statements made herein of my knowledge are true and that all statements made on information and belief are believed to be true and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment or both under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Date: October 25, 2007

A. Moravsky

Alexander P. Moravsky

RESUME
ALEXANDER P. MORAVSKY

EDUCATION

Ph.D. Chemistry, Moscow Institute for Physics and Technology (MIPT), 1975
M.Sc. Physics of Fast Processes, MIPT, 1972
B.Sc. Molecular and Chemical Physics, MIPT, 1970

PROFESSIONAL EXPERIENCE

1998-Present MER Corporation, Senior Scientist, Tucson, AZ
1980-1998 Senior Research Fellow, Head of a Research Group, Institute of Problems of Chemical Physics, Chernogolovka, Russia
1979-1980 Post Doctorate of Prof. J.K.Stille, Colorado State Univ., Fort Collins, CO
1975-1979 Research Fellow, Institute of Chemical Physics, Chernogolovka, Russia

RECORD OF TECHNICAL ACHIEVEMENTS

- 11 Patents
- 230 papers in refereed journals
- 5 Reviews

INDUSTRIALLY IMPLEMENTED TECHNOLOGIES:

- technique for ketones and aldehydes micro-analysis
- methane absorption accumulators

AREAS OF TECHNICAL EXPERTISE

- R&D on advanced new materials, basic chemical technology, organic synthesis
- Catalysts, small molecule catalytic conversions of industrial interest
- Redox processes and fast reactions in solution and films
- Purification of substances, analytical chemistry
- Analysis of sophisticated chemical kinetic schemes
- Spectral analysis, NMR and ESR, XPS, GC, GC/MS, LC, HPLC
- Fullerene and nanocarbon materials science and technology

Dr. Moravsky A.P.
List of recent publications. 1993-2007.

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"Photochemical Properties of C_{60}. Triplet Excited C_{60} Quenching by Electron Acceptors TCNQ and TCNE in Solution. Laser Photolysis Study", Chem.Phys.Letters, v.208, No5-6, 431-435 (1993), V.A.Nadtochenko, N.N.Denisov, I.V.Rubtsov, A.S.Lobach, and A.P.Moravskii.

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"Photochemical Properties of C_{60}. Photooxidation of C_{60} by TCNE and TCNQ in Solutions. Laser Photolysis Study". Fullerenes. The First Int. Interdisciplinary Colloquium on the Sci. and Tech. of Fullerenes, 1993, Santa Barbara, USA, Abstract, p.192-194. Nadtochenko V.A. et al.

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"On the Reaction of Buckminsterfullerene with Tetrabuthylammonium Tetrahydroborate", Russian Chemical Bulletin, N3, 803(1993). V.D.Makhaev, Yu.M.Shulga, A.S.Lobach, V.N.Vasilets, O.S.Roshchupkina, and A.P.Moravsky.

10. "Reaction of Organic Cation Tetrahydroboronates with Fullerene C_{60}", Int. Conference "Physics and Chemistry of Fullerenes", Greece, 1993, Abstracts, p.34. V.D.Makhaev, A.S.Lobach, Yu.M Shulga. O.S.Roschupkina, and A.P.Moravsky.

"Interaction of Methane with Acetylene in the Presence of Ziegler-Natta Type Catalysts", Neftekhimiya, v.32, N4, 324-330 (1992). E.M.Efimova, A.P.Moravskii, and N.F.Noskova.

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"Picosecond Dynamics of Charge Recombination in EDA Complexes between C_{60}, C_{70} and ternary Amines", IW Fullerenes and Atomic Clusters, 1995, p.109-110, N.N.Denisov, I.V.Rubtsov, A.S.Lobach, A.P.Moravsky, and V.A.Nadtochenko.

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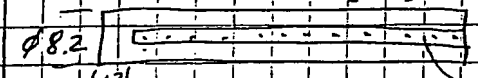
Run CFS9

H_2 175 Torr (600 sccm) + Ar 125 Torr (200 sccm) = 350 Torr

Rod: ℓ_{202} (8") I 81 A v 2.0 mm/min



(13)

Chim Cat Composition: 51.05% Ni, 13.8 Co, 27.1 Fe, 8.05 S

21.81 g
16.66
5.15 g (4:1 = M : Graph)
↳ 4.12 g M + 1.03 g C

Steady-state vaporization found at U 26÷28 V at I 81 ÷ 82.0

Composition of the rod:

$\Sigma C = 16.65 + 1.3 + 1.03 = 16.4$ g ; $\Sigma C + M = 20.52$ Chung

Ni = 2.103 g	$\Rightarrow \frac{2.103}{16.4}\cdot\frac{12}{58.69}$	= 2.62 at% = 10.26 wt%		2.6
Co = 4.12 · 0.138	$\Rightarrow$	= 0.706 at% = 2.77 wt%		0.7
Fe = 4.12 · 0.271 = 1.116		1.46 at% = 5.44 wt%		1.45
S = 4.12 · 0.085 = 0.353		0.76 at% = 1.62 wt%		0.75

Total burning 45 min.

Product: 1.17 g WS : pWS 0.45 g MG/B : passes sieve
0.22 g sieved powder
Deposit + Ynk 12.4 g ? Left: crispy powder, burned webs
Sieves 2.8 g
Rod Remainder 1.6 g

SEM pWS : Curly bundles ϕ ~ 100 nm embedded in amorph, very long (> 5÷10 μm)

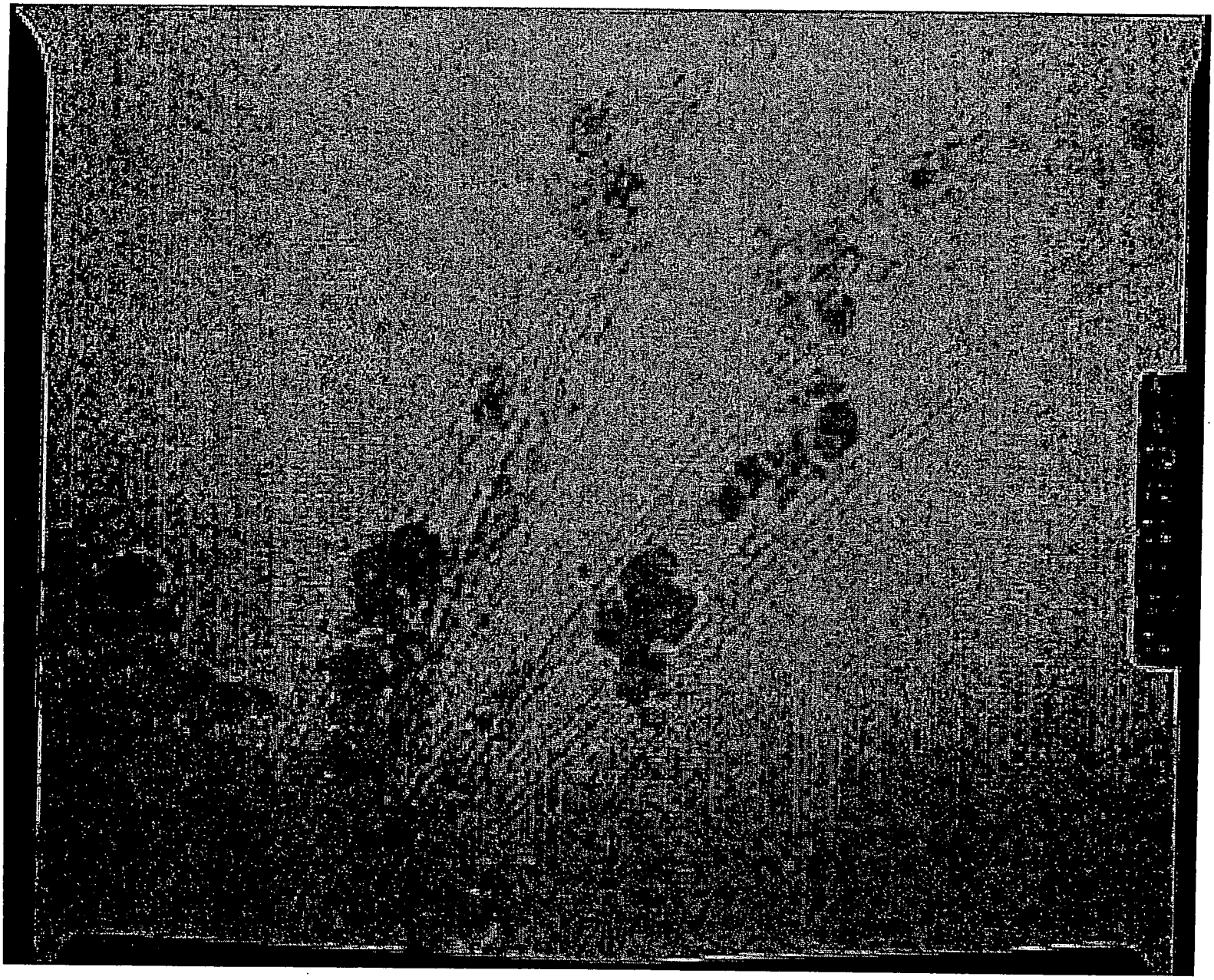
TEM : Thin bundles of 5–20 tubes & some separate thick tubes of ~ 4–5 nm ϕ
Most of tubes have 2 walls!
Some are SWNTs (thick ϕ ~ 4÷5 nm)
(The system selectively produces double-walled nanotubes in abundance!)

A. Moravsky

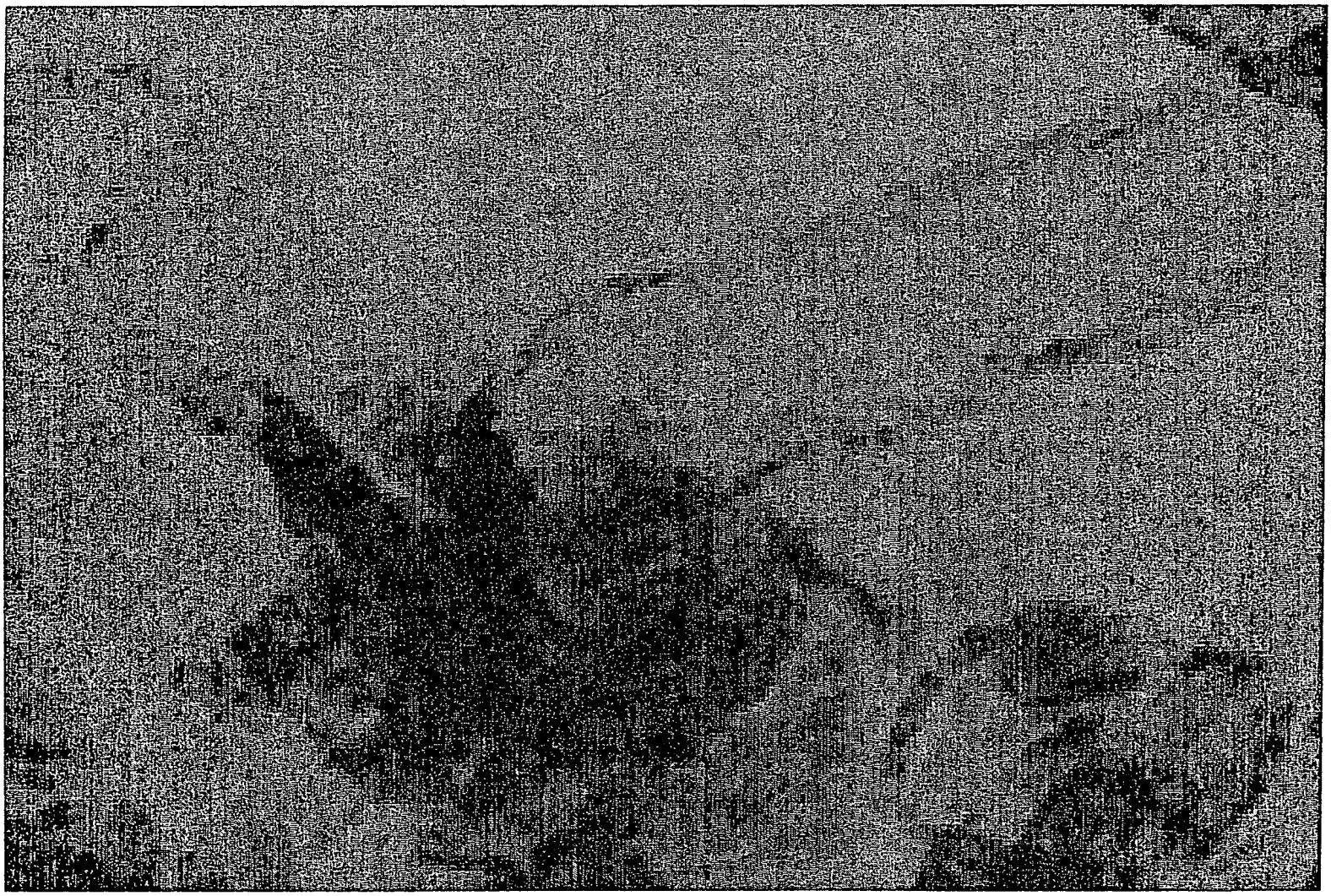
Read & Understood

Howe

TEM image of the product of Run CF39



TEM image of the product of Run CF39



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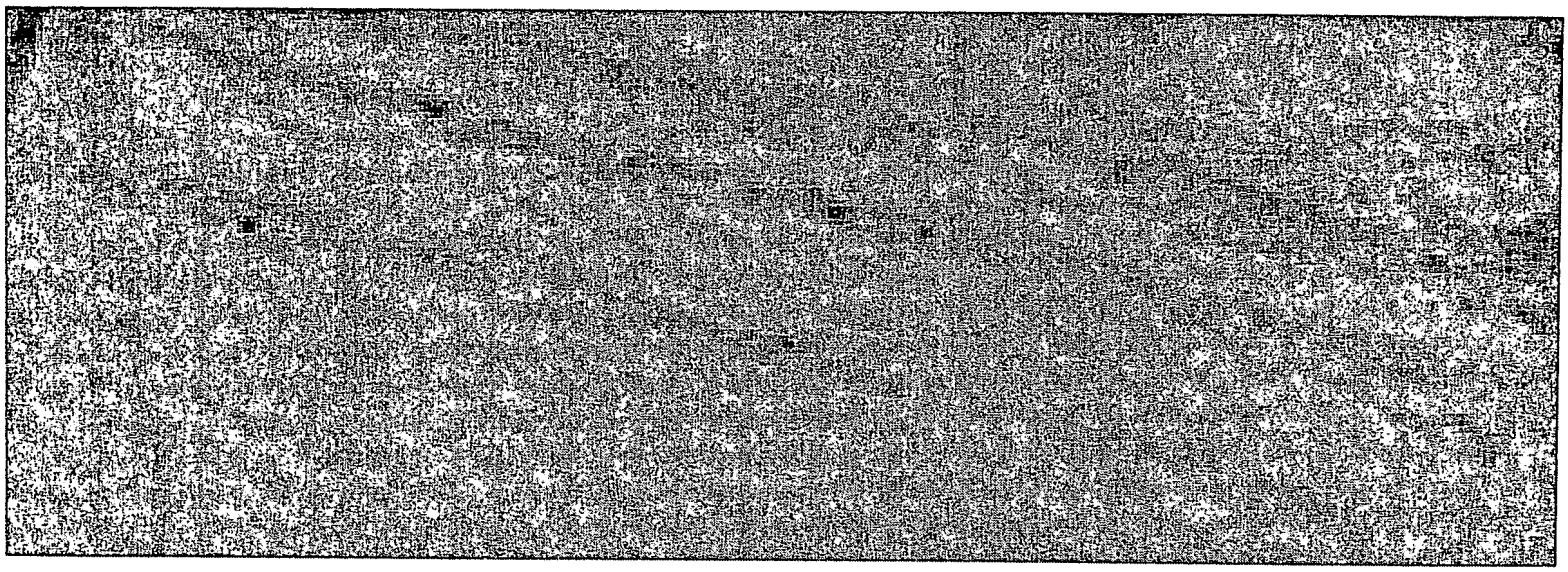
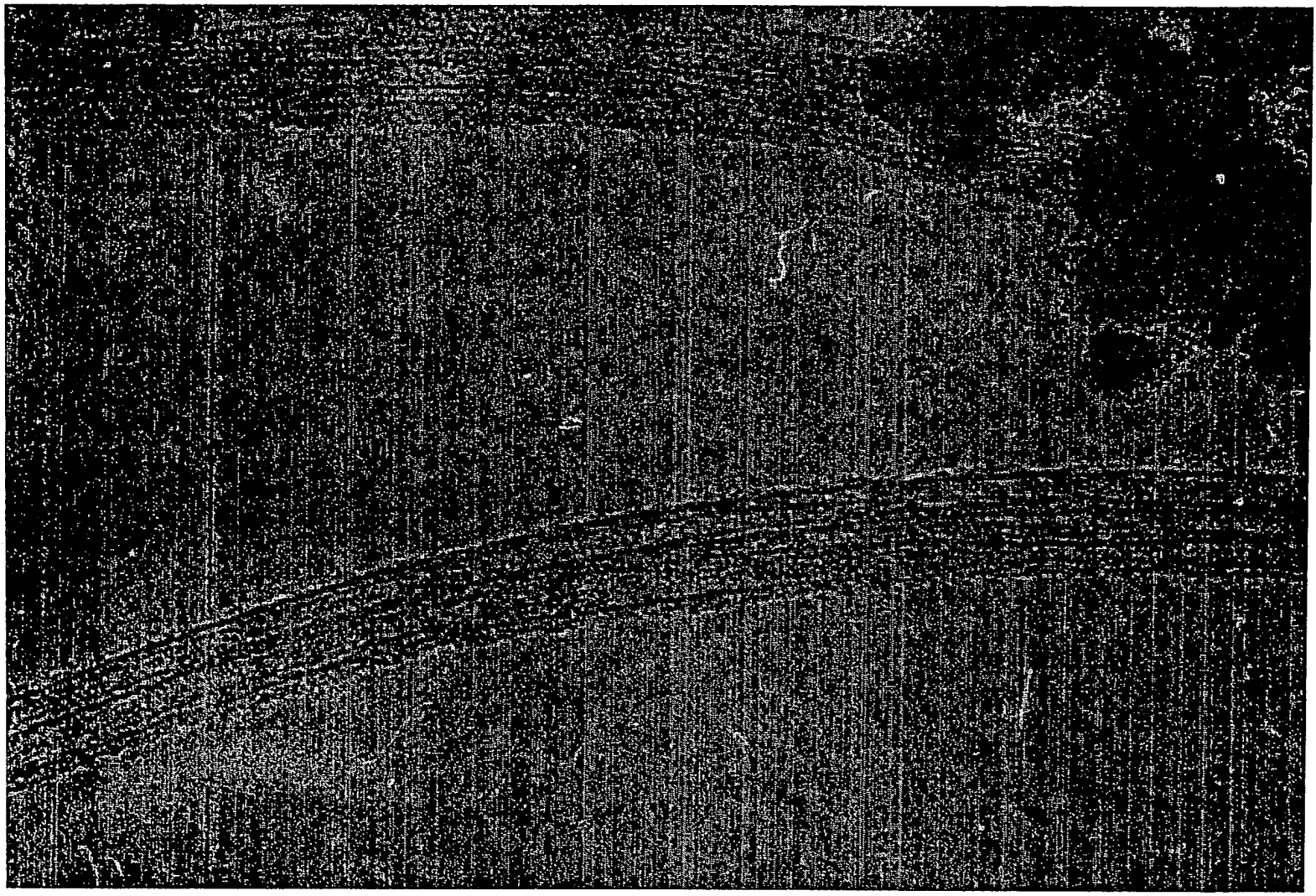
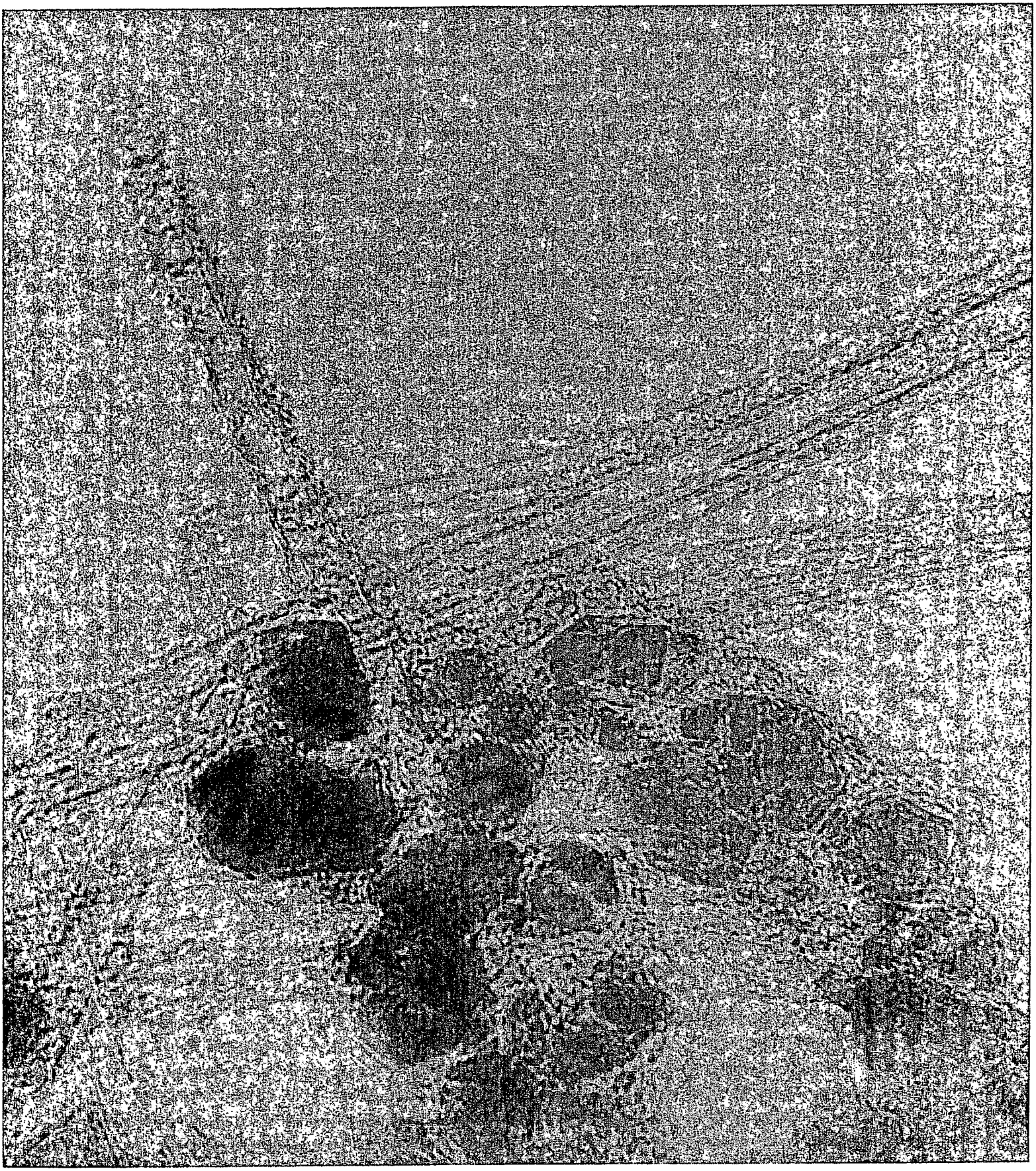


TEM image of the product of Run CF39

TEM image of the product of Run CF39



TEM image of the product of Run CF39



Subject: Message for Dr.A.P.Moravsky
Date:
From: "Elena P. Krinichnaja" <elkrina@icp.ac.ru>
Organization: IPCP
To: "moravsky@Opus1.com" <moravsky@Opus1.COM>

Саша, здравствуй!
ТЭМ (кобальт, никель, железо и сульфид железа): двухслойные нанотрубки, диаметр 3.5-5.2 нм, расстояние между стенками 0.42+/-0.02 нм.
Внутренние каналы нанотрубок частчно заполнены, по всей видимости , аморфным углеродом. Помимо нанотрубок и каталитических частиц в образце присутствуют различные формы углерода, структура которых на данный момент времени не анализировалась.
Елка..
П.С. Ты когда-нибудь ответишь мне?

E-mail message for Dr. A.P.Moravsky from Dr.E.P.Krinichnaja

Translation into English

Hello, Alex!

TEM (cobalt, nickel, iron and iron sulfide catalyst) has shown: double-walled nanotubes of 3.5 – 5.2 nm diameters, with the distance between the walls 0.42 +- 0.02 nm. Internal channels of nanotubes are partially filled, most likely, with amorphous carbon. Besides the nanotubes and catalytic particles, there are present various forms of carbon with the structure that was not yet analyzed.

Elena.

P.S. Waiting for your reply.

PERGAMON

Carbon 39 (2001) 761–770

CARBON

Double-walled carbon nanotubes fabricated by a hydrogen arc discharge method

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Abstract

Double walled carbon nanotubes (DWNTs) were obtained by the arc discharge technique in an atmosphere of Ar and H_2 mixture (1:1/v:v) at 350 Torr. The catalyst was prepared from a mixture of Ni, Co, Fe and S powders heated in an inert gas atmosphere at 500°C for 1 h. High resolution electron microscopy (HREM) revealed that the dominant type of obtained nanotubes were DWNTs with outer diameter in the range of 1.9–5 nm and inner tube diameters in the range 1.1–4.2 nm. As a rule, the DWNT tubes form into bundles. Occasionally single walled nanotubes (SWNTs) were observed by HREM although Raman spectroscopy did not reveal the presence of significant quantities of these tubules in the bulk product.

Keywords: A. Carbon nanotubes; B. Plasma deposition; C. Transmission electron microscopy; Raman spectroscopy; D. Microstructure

1. Introduction

Selective production of specific types of carbon nanotubes (NTs) is one of the principal goals of NT synthesis technology. This is necessary for establishing the correlation between different types of physical properties (electro- and heat conductivity, mechanical rigidity and so on) and the obtained NT type, for testing the validity of the theoretical predictions regarding their respective properties and also for the achievement of the specified properties within bulk purified samples.

The synthesis of single-walled NTs (SWNTs) in an atmosphere of hydrogen by arc discharge techniques was recently published [1,2]. As a catalyst, a mixture of Ni, Co, Fe and FeS was evaporated together with the carbon feedstock. The reactor construction was modified relative to the conventional arc discharge method normally used for SWNTs production [3–5]. Instead of a catalyst-filled anode, a set of round grooves on the anode surface was filled with a mixture of carbon and the catalyst powder. The cathode axis was also oriented at an angle to the anode axis instead of the 'end to end' anode–cathode geometry normally employed. In these conditions, high quality SWNTs were obtained. The specimens were investigated by resonant Raman scattering measurement and by HREM. Three well-resolved peaks at 1.593 cm^{-1}, 1.576 cm^{-1} and 1.569 cm^{-1}, can be assigned to a splitting of the E_{2g} mode of graphite considered should be characteristic of SWNTs [6]. HREM observations revealed that, for the SWNT bundles, the mean SWNT diameter was 1.85 nm [1].

We performed experiments in conditions very close to those in Ref. [1], except that the normal configuration of

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PII: S0008-6223(00)00187-1

electrode construction and orientation (i.e. 'end to end') was used. Under these conditions, as we describe here, DWNTs were formed as the dominate component in the product mixture.

2. Experimental

2.1. Preparation of nanotubes

To produce NTs, arc discharge evaporation of a metal–graphite electrode was used. The anode was a graphite rod (8.2 mm in diameter) with a drilled channel (3.2 mm in diameter and 140 mm in length) filled with catalyst. The cathode was a graphite rod 10 mm in diameter and 25 mm in length. The catalyst was prepared in the following way: a mixture of Ni, Co and Fe powder metals (particle sizes 2–5 μm) and elemental S was ground in a mortar and then heated in an inert gas atmosphere at 500°C for 1 h. The conglomerate was then ground in a ball mill to a micron particle size and then intimately mixed with carbon powder. A 3.2-mm channel drilled in the graphite anode was tightly filled with this mixture. The content of elements in the composite anode with respect to C was: Ni 2.6, Co 0.7, Fe 1.45 and S 0.75 at.% and so matched the metal and sulfur content of the anode materials used for the synthesis described in Ref. [1].

The process of catalyst synthesis was carried out with an arc current of 75–80 A in an atmosphere of Ar and H_2 mixture (1:1/v:v) at 350 Torr with a 410-Torr partial vacuum. The voltage drop across the gap was 26–28 V and the gap width kept constant and equal to 2 mm. The duration of the arc evaporation process was about 40 min. Two types of carbon material were obtained: (1) the product deposited on the reactor walls; and (2) the remaining product deposited on electrodes and cathode deposit as an elastic web-like product.

2.2. Electron microscopy

Specimens for TEM and HREM were prepared by dispersing the web-like products collected from the chamber wall and between the electrodes in acetone in an ultrasonic bath. A drop of the suspension was put on a lacy carbon film supported by a copper grid. Specimens were investigated in a Philips EM-430ST electron microscope operated at 200 kV, in a JEOL JEM-4000EX operated at 400 kV and JEM-3000F instrument operated at 300 kV with Oxford Instrument ISIS 300™ EDX system and Gatan (1 k×1 k) CCD camera. To calibrate the magnification in the EM-430ST, lattice fringes obtained from Si crystals in [110] orientation were used as a reference standard.

2.3. Raman spectroscopy

Raman measurements were performed with a Jobin Yvon S-3000 spectrometer equipped with a triple monochromator and operated in microRaman configuration. Ar^+-laser irradiation with a wavelength of 514.5 nm was used for spectra excitation. Since the material was very sensitive to heating, the measurements were carried out at low laser power (i.e. less than 20 mW).

3. Results

A general view of the specimen is shown in Fig. 1a. The nanotubes form readily into bundles with discrete tubules clearly visible on the periphery. The observed length of these tubes in bundles is more than 1 μm. The HRTEM micrographs (Fig. 1b) revealed that most of the tubules have a specific structure and consisted of two concentric cylindrical graphene layers, that is they are double wall nanotubes (DWNTs). In general, the DWNTs were measured with outer diameters of between 1.9 and 5 nm while the diameter of the inner tube varied between 1.1 and 4.2 nm. Along with the bundles, discrete separated DWNTs were also observed. These discrete DWNTs are easily distorted (Fig. 2). That means they are mechanically less rigid in comparison with single wall nanotubes (SWNTs), probably because of their larger diameter. The tubule diameter in Fig. 3a varied between 3 nm and 4.95 nm. For the DWNT in Fig. 3b, a variation of the tubule diameter from 3.75 to 5.0 nm along the length of the tube is visible at intervals of between 30 and 35 nm. Some DWNTs have rounded double layered terminating caps at the end (Fig. 3c). Notably, the interlayer distance measured in the tubule wall images taken at 200 kV and 400 kV is 0.39±0.02 nm which is larger than that usually observed for multilayered NTs (0.34 nm). A micrograph of a DWNT obtained in the 3000F microscope and, from the indicated wall region, contrast density profile are shown in Fig. 6. The latter reveals an interlayer separation of 0.403 nm. A range of similar measurements obtained from other DWNTs revealed that this wall spacing was typical.

Often catalytic particles (CPs) surrounded by one to several graphene layers were observed (see Fig. 1b, Fig. 2 and Fig. 4) with interlayer distances of the order of 0.36±0.02 nm. HRTEM images of CP vary in lattice images. There is indirect evidence that during the formation of the DWNTs, the CPs are in the melt state since some DWNTs are filled with dense material (Fig. 3d). EDX spectrum from CP revealed peaks corresponding to Co, Ni and Fe. Traces of S were also revealed (see, for example Fig. 5).

In some DWNTs, fragments of third and fourth (Fig. 3a) layers were observed. At the same time, SWNTs were seldom observed (Fig. 4). Their diameters were in general found to be close to the inner tube diameter in the observed DWNTs (i.e. 2.7–4.7 nm). It is believed that such SWNTs could be considered as DWNTs in which the formation of the second layer does not occur or was

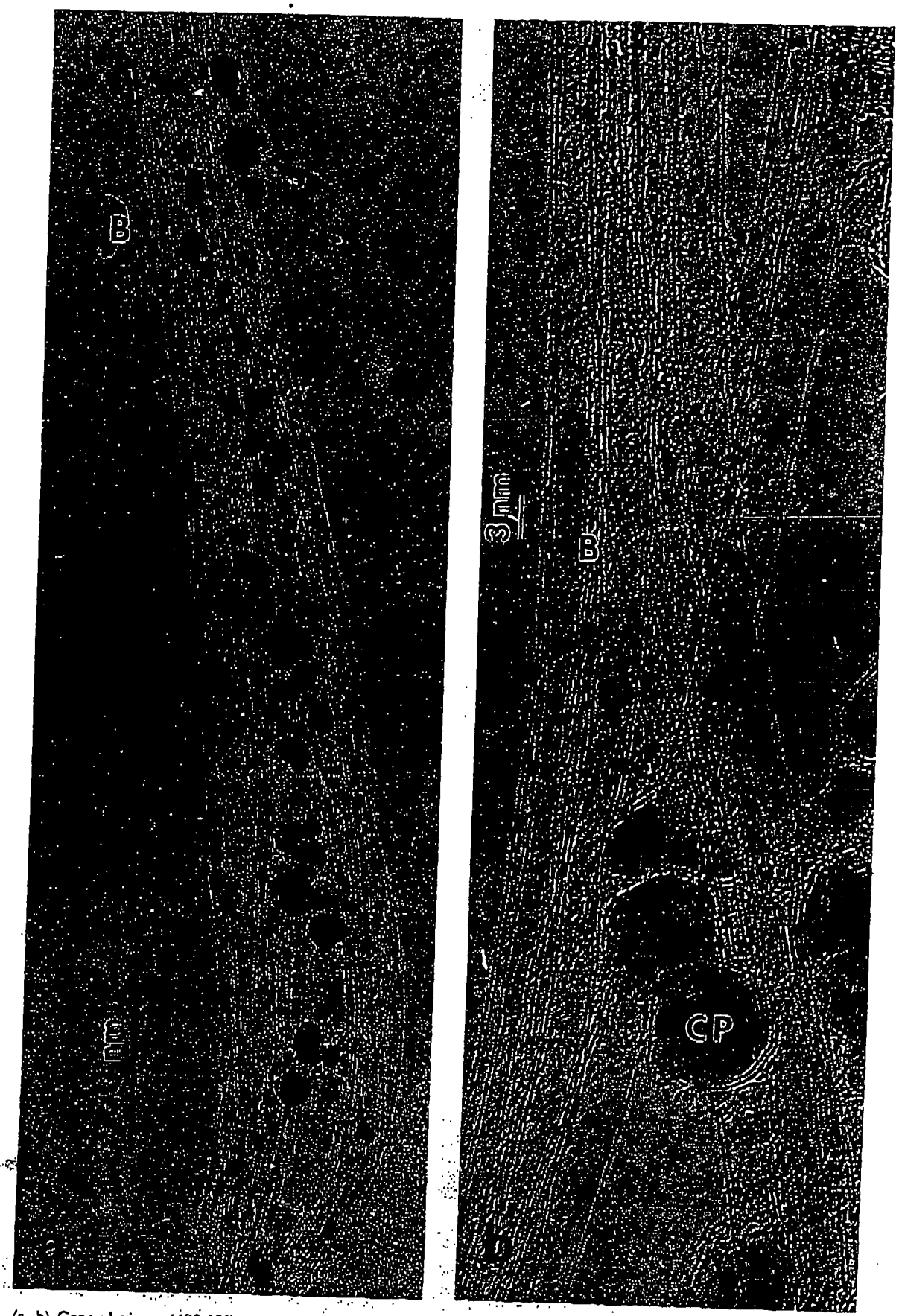

Fig. 1. (a, b) General views (400 kV) showing bundles (B) of straight double walled nanotubes (DWNTs): CP, catalytic particle.

Fig. 2. General view (400 kV) showing bundles (B) and separate DWNTs. The regions of DWNT distortion are marked by large arrows. Small local variations of nanotube diameter are marked by small arrows.

incomplete. A few NTs were observed that conformed to small diameter (i.e. 1.7–2 nm) SWNTs.

Along with DWNTs diameter, variations were caused by tube deformations resulting, in some cases, in local small variations (10–15%) in diameter. These variations are sometimes observed only for inner tubes (Fig. 2 and Fig. 4). They are visible in SWNT images as well (Fig. 6). In our opinion local variations are connected with the process of nanotube growth.

The Raman spectrum (Fig. 7) was dominated by the

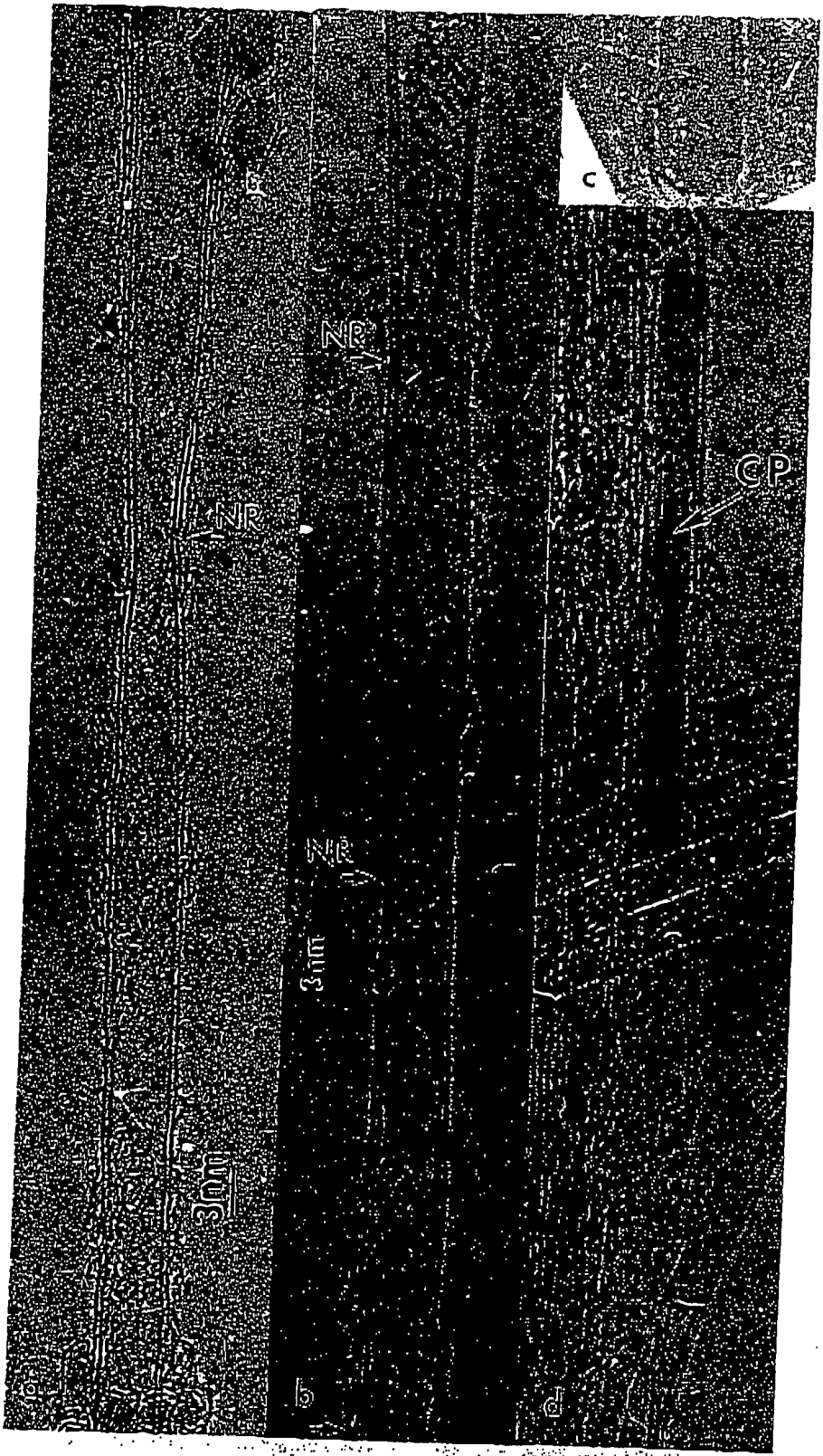

Fig. 3. Separate nanotubes of different types: (a) DWNT with varying diameter (400 kV). Narrow region (NR) is arrowed. Small fragments of the third and fourth layers are visible (marked by large arrows). (b) DWNT with pseudoperiodically varying diameter (200 kV). Several narrow regions (NR) are arrowed. Some forms of amorphous carbon material are observed both inside and outside the tube. (c) DWNT with rounded end cap (200 kV). (d) DWNT filled with dense material, probably CP (200 kV).

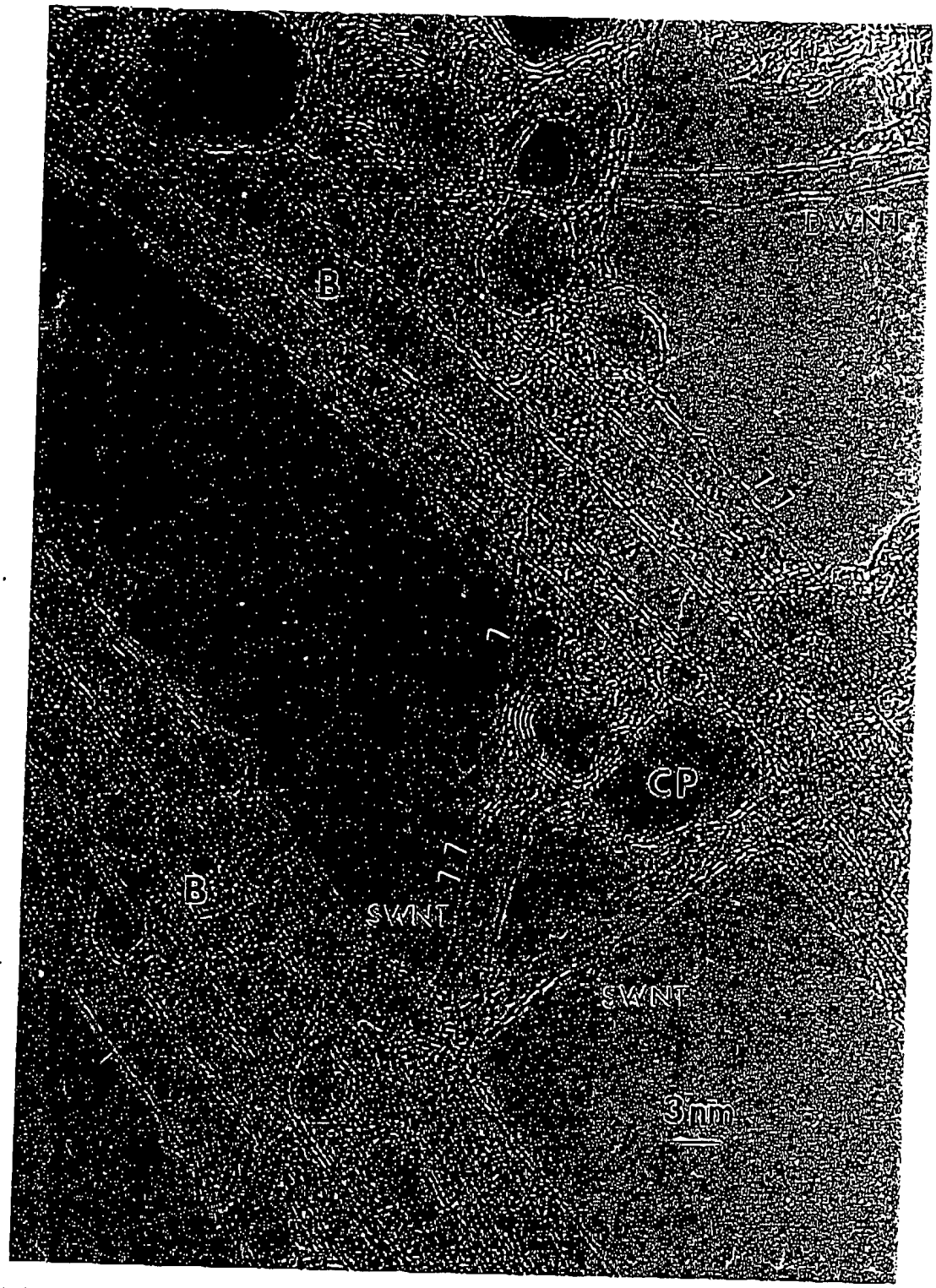

Fig. 4. General view of DWNTs (400 kV) showing bundles (B), separate DWNT with relatively small diameter (2.2–2.5 nm) and SWNTs. Small local variations in the DWNT and SWNT diameters are marked by small arrows.

band at 1582 cm^{-1} with a full width at half maximum (FWHM) of 50 cm^{-1}. This feature may be assigned both to well-crystallized graphite [7] and to multiwall carbon nanotubes [8]. Unfortunately there are no characteristic 'fingerprint' features for multiwall carbon nanotubes in the Raman spectrum which enables them to be distinguished from graphitic carbon in the Raman spectrum. This is the case only for single-wall carbon nanotubes which demonstrate a splitting of the tangential mode (i.e. near 1592 cm^{-1}) and an appearance of the 'breathing' mode with a

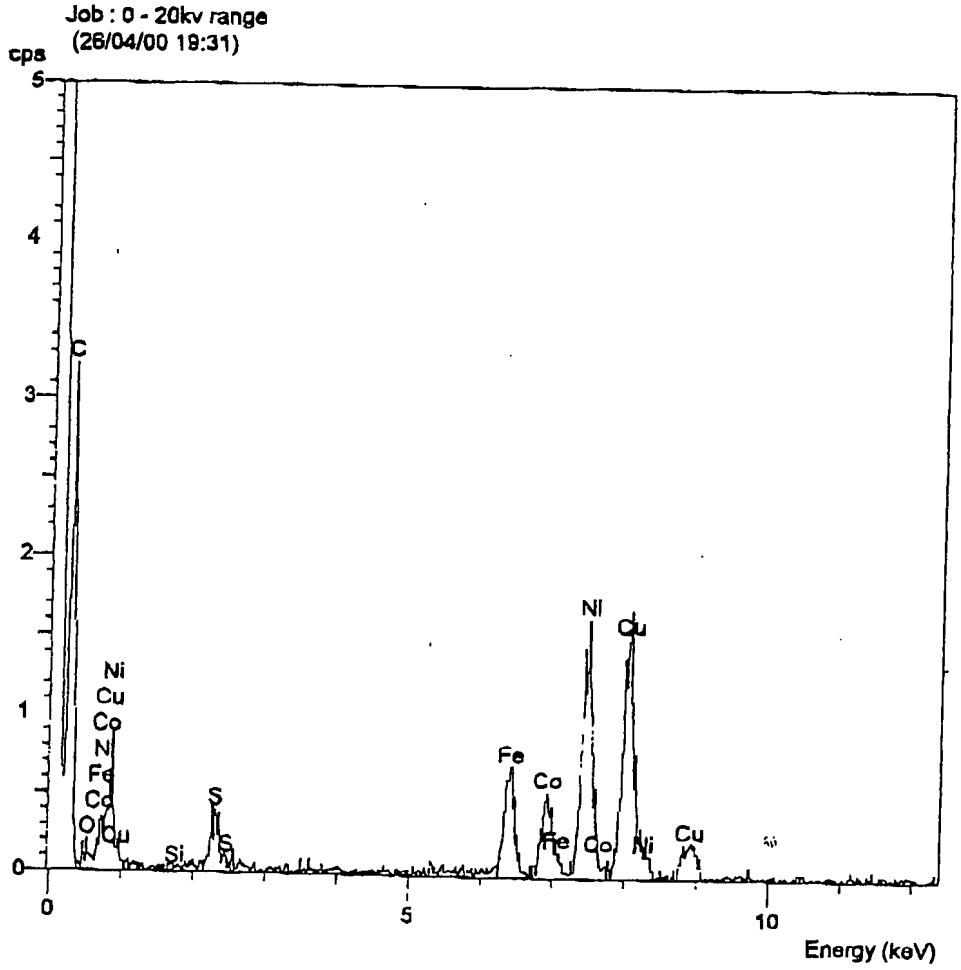

Fig. 5. EDX spectrum.

frequency strongly depending on the tube diameter in the range 100–200 cm^{-1} [9,10]. These features are observed for nanotubes with a diameter less than 2 nm [11]. For larger nanotubes they disappear due to overlapping of maxima in the nanotube electronic density.

4. Discussion

The dominant type of structure found in our product were DWNTs. Within the observed tubules, there was a clear distribution of diameters suggesting that a number of different (*n,m*) conformation nanotubes were present in the inner and outer tubules making up the composite DWNT tubules. A few nanotubes were observed with third and fourth layers but these were usually incomplete or damaged. This phenomenon could possibly be accounted for by the employed experimental conditions (for example the presence of hydrogen).

As mentioned above, a small number of SWNTs were observed in the specimen and their minimum diameter was found to be ca. 1.7 nm. The small quantity of these nanotubes observed within the specimen also accounts for the absence of the corresponding peaks within the Raman spectrum. Peaks corresponding to SWNTs were reported in Ref. [1] in which a far higher proportion of SWNTs was observed in the product. It might have been expected that for the DWNTs the signals from both shells would have been seen in the Raman spectrum. However in the case of our product the average tube diameter was too large to observe this effect.

It has been shown that addition of sulfur to the starting mixture of C and Co significantly increased the yield of SWNTs found in the arc discharge product [12]. In addition a greater spread in the size distribution of the observed SWNTs was reported and, occasionally, single DWNTs were observed. It was suggested that sulfur may affect the kinetics by removing terminating species from the growing ends of the tubes. Another possibility is that sulfur species could bind to and stabilize the ends of large diameter tubes. In the modified experiment described here the catalytic arc-discharge system has selectively produced DWNTs as the main product collected from electrodes in the reactor (although not from the cooled walls).

It is necessary to mention that even the smallest deviation from the synthesis conditions described above

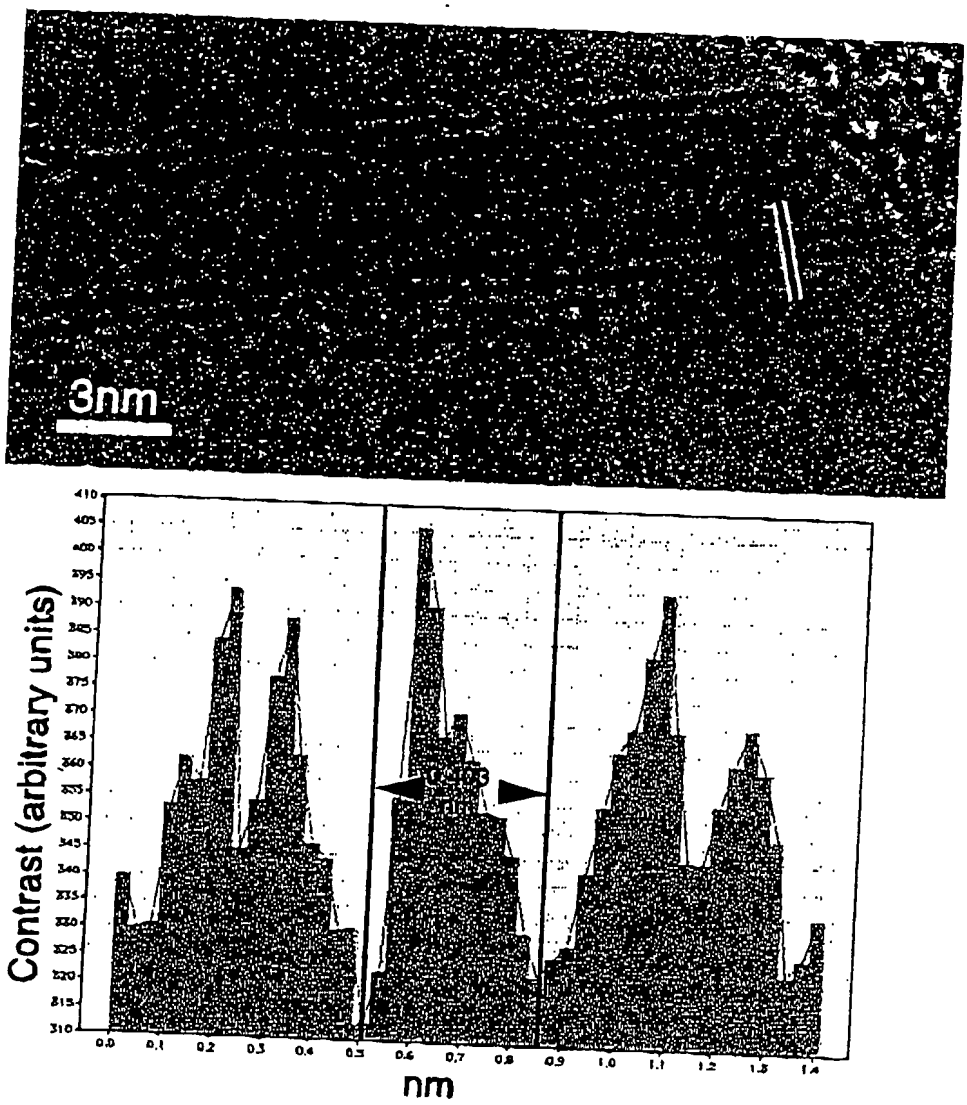

Fig. 6. DWNT micrograph (top) obtained in a JEM-3000F microscope 300 kV and a contrast density profile (bottom) of the nanotube wall, revealing an interlayer distance 0.403 nm.

led to a decrease in the formation of DWNTs and resulted in a domination of SWNTs in the product mixture. In general the amount of DWNTs in the product varied from ~10–20 wt.% to ~50–70 wt.%. It appears therefore that the conditions and arcing geometry of the experiment are close to optimal for the selective and effective production of DWNTs via the arc discharge method.

To optimize the process of SWNTs synthesis, a series of experiments in an arc-discharge reactor was performed with a wide range of Co:Ni:Fe catalyst compositions with controlled arc-discharge parameters [13]. In all cases SWNTs were produced in the synthesis product and only a very few DWNTs were observed. This begs the question: what has drastically changed the morphology of the condensation products in the conditions used in the present paper? To clarify this we performed additional experiments that showed that sulfur addition to the metallic catalyst when the arc-discharge process was carried out only increased the SWNTs yield, but did not lead to the formation of DWNTs. This finding is in good agreement with published results on the influence of sulfur on the catalytic synthesis of carbon nanotubes. It was shown previously [12] that sulfur addition to carbon and cobalt mixture led to an increase in the yield of SWNTs. In our case, sulfur addition resulted in an increase in the nanotube diameter distribution (1–6 nm).

We have also performed experiments to reveal the role of hydrogen. It was shown that in the products formed from arc discharge in H_2/Ar atmosphere DWNTs appeared and even predominated. This indicates that the presence of sulfur in the catalyst is not critical for the formation of DWNTs. In order to understand DWNTs appearance when He atmosphere is replaced by a mixture of Ar and H_2, it is necessary to explain how the process is changed.

It is known that at similar arc parameters (i.e. current and gas pressure) the carbon plasma temperature in Ar is

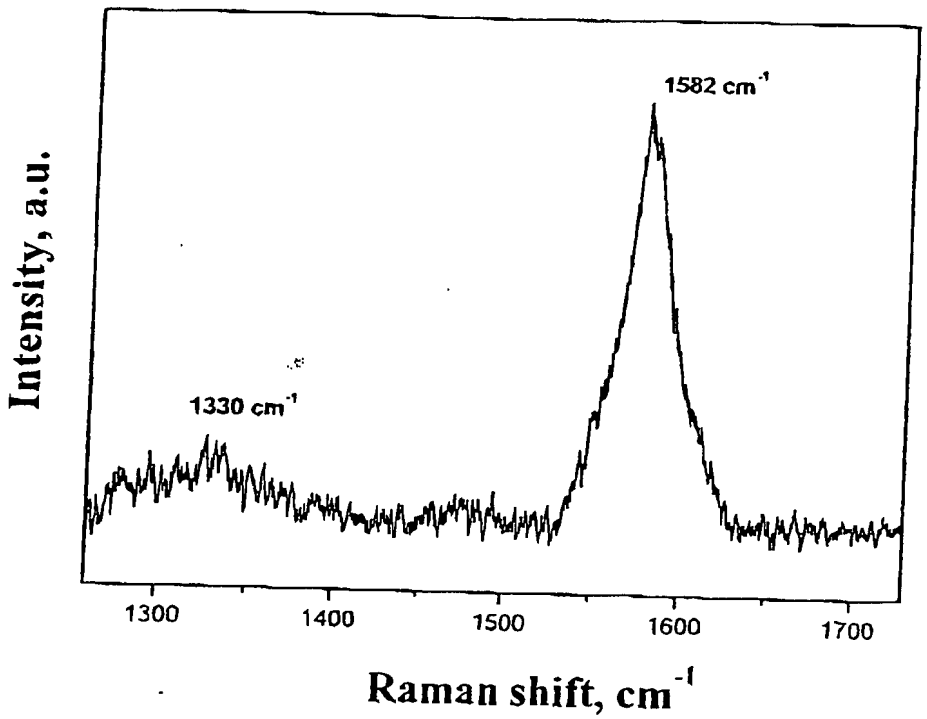

Fig. 7. Raman spectrum of the DWNTs product.

essentially lower than in the He atmosphere. The anode carbon in Ar is evaporated only slowly during arcing. Apparently, this is due to the larger value of the He ionization energy (24.5 eV) compared to the Ar one (15.76 eV). This means that a higher plasma temperature is required in case of He in order to produce the same equilibrium concentration of current carriers. If the helium arc temperature is 3600–3900 K then this is sufficiently large for carbon evaporation [14]. The Ar arc temperature in the same conditions can be approximated to 2400–2600 K which is indeed not hot enough for anode graphite evaporation [15]. However if H_2 is added to the Ar atmosphere, the anode evaporation is very rapid. The gas temperature in the arc in this case does not change relative to that obtained in pure argon. This means that the anode graphite, when H_2 is added, is evaporated in the form of hydrocarbon fragments, radicals and molecules. Therefore, under these conditions, the arc-discharge process is transformed into a gas-phase analog of a catalytic pyrolysis process with a 'floating' catalyst.

The complete pattern of the process of carbon formation in the arc-discharge reactor when H_2 is added is obviously more complicated in comparison with that which occurs in an inert atmosphere. At present, not all of the details are clear, but some phenomena are interpretable. For example, from the available thermodynamic data it is possible to estimate that, under the present conditions of evaporation (2400–2600 K), the equilibrium concentrations of polyyne radicals of the form $C_{2n}H^{\cdot}$, with $n=1, 2, \ldots$ are only a small fraction of the overall polyyne concentrations itself. The reason for this is the large energy of the C–H bond in the polyyne molecule, which is ~120 kcal/mol. Therefore, in an arc gas and in an Ar and H_2 atmosphere at 2400–2600 K the large proportion of carbon clusters is in the form of acetylene and polyyne molecules of the form $C_{2n}H_2$.

If one temporarily puts aside the role of the catalyst and the influence of the arc discharge atmosphere then, structurally, the different types of NTs can be aligned in the following series in accordance with synthesis temperature: SWNTs>MWNTs>CLNTs>NFs (NFs=nanofilaments). According to synthesis temperature DWNTs must be located somewhere between MWNTs and SWNTs. It is noticeable that the thermal conductivity of NTs is decreased passing from SWNTs to CLNTs and NFs.

Acknowledgements

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